

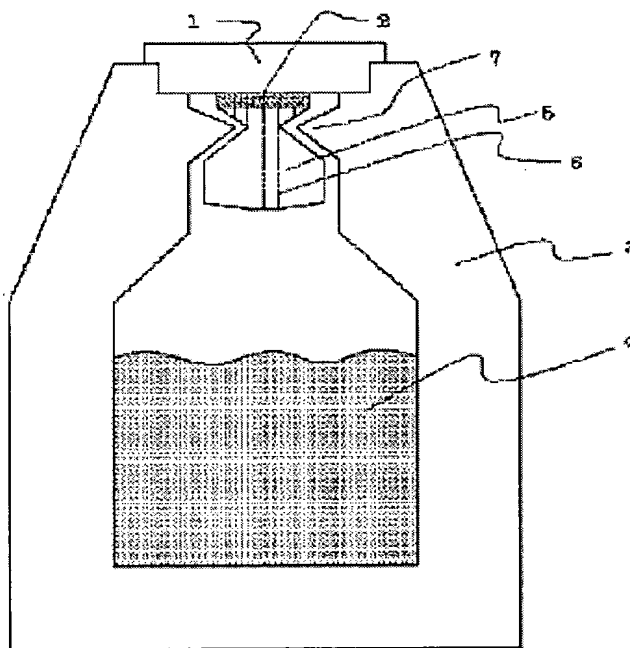
**PRODUCTION OF SINGLE CRYSTAL SILICON CARBIDE**

**Patent number:** JP5319998  
**Publication date:** 1993-12-03  
**Inventor:** OTA KIYOSHI; KOGA KAZUYUKI  
**Applicant:** SANYO ELECTRIC CO  
**Classification:**  
**- international:** *C30B23/02; C30B29/36; C30B23/02; C30B29/10;*  
(IPC1-7): C30B29/36; C30B23/02  
**- european:**  
**Application number:** JP19920148835 19920515  
**Priority number(s):** JP19920148835 19920515

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**Abstract of JP5319998**

**PURPOSE:** To provide a method for growing a single crystal of good quality with hardly any defects. **CONSTITUTION:** Protrusions 7 are formed near a seed crystal 2 of a crucible 3 for growing a crystal, which is once slenderly narrowed with the protrusions, grown and reenlarged to grow a silicon carbide single crystal 6 in a method for thermally subliming a raw material 4 composed of silicon carbide, feeding the sublimed raw material onto the seed crystal 2 composed of the silicon carbide single crystal and growing the silicon carbide single crystal 6 on the seed crystal 2.



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**JAPANESE** [JP,05-319998,A]

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CLAIMS DETAILED DESCRIPTION TECHNICAL FIELD PRIOR ART EFFECT OF THE INVENTION  
TECHNICAL PROBLEM MEANS OPERATION EXAMPLE DESCRIPTION OF DRAWINGS DRAWINGS

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**CLAIMS**

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**[Claim(s)]**

[Claim 1] The manufacture approach of the single crystal silicon carbide which is made to carry out heating sublimation of the raw material which consists of silicon carbide, and is characterized by supplying on the seed crystal which consists of a silicon carbide single crystal, once extracting a crystal thinly, making it grow up in the approach of growing up a silicon carbide single crystal on this seed crystal, and expanding again.

[Claim 2] The manufacture approach of the single crystal silicon carbide according to claim 1 characterized by preparing a projection near the seed crystal in the crucible for crystal growth, and once extracting a crystal thinly.

[Claim 3] The manufacture approach of the single crystal silicon carbide according to claim 1 characterized by establishing the means which makes an elevated temperature near the seed crystal in the crucible for crystal growth, and once extracting a crystal thinly.

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DETAILED DESCRIPTION

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[Detailed Description of the Invention]

[0001]

[Industrial Application] This invention relates to the manufacture approach of a silicon carbide single crystal, and relates to the approach of growing up a good single crystal with few defects especially.

[0002]

[Description of the Prior Art] Silicon carbide (SiC) is excellent also in thermal resistance and a mechanical strength, and attracts attention as an environment-resistant semiconductor material from physical and chemical property, like it is strong in a radiation. At the room temperature, especially the SiC crystal of 6H form has forbidden-band width of face of about 3eV, and is used as a blue light emitting diode ingredient.

[0003] As an approach of growing up the ingot of such a SiC single crystal, the sublimating method is adopted, for example as indicated by "a vacuum and 30 pages [ 52-58 ] (1987)."

[0004] The sectional view of the crucible of the conventional growth equipment used for the sublimating method at drawing 3 is shown. The crystal growth of the conventional SiC puts the SiC powder 4 as a raw material into the lower part of the crucible 3 by which it is covered with the electrode holder 1 which fixed the seed crystal 2 which consists of a 6 H-SiC single crystal, for example, heats a crucible 3 in it by the RF creeping method of adjustment, makes the above-mentioned powder SiC4 sublimate to it, and supplies a gas raw material on the above-mentioned seed crystal 2. And the raw material of a gas [ making the above-mentioned seed crystal 2 into the low-temperature section ] recrystallizes, and a single crystal 5 grows on the above-mentioned seed crystal 2.

[0005] Thus, as shown in drawing 3 R> 3, many line defects 6 exist in SiC by which crystal growth was carried out. It seems that in addition, the line defect 6 shown in drawing 3 R> 3 is shown typically, and is not in sight in practice with the naked eye.

[0006]

[Problem(s) to be Solved by the Invention] When growing up a single crystal by the \*\*\*\* conventional approach, the 104 about [ /cm / 2 ] consistency of a defect existed, and these defects had difficulties, such as having an adverse effect on the electrical characteristics of light emitting diode (LED).

[0007] This invention is made into what was made in view of the above-mentioned situation, and sets it as that object to offer the approach of growing up a good single crystal with few defects.

[0008]

[Means for Solving the Problem]

[0009] The manufacture approach of the single crystal silicon carbide this invention carries out heating sublimation of the raw material which consists of silicon carbide, it is supplied on the seed crystal which consists of a silicon carbide single crystal, once extracts a crystal thinly, makes it grow up in the approach of growing up a silicon carbide single crystal on this seed crystal, and is characterized by expanding again.

[0010]

[Function] According to this invention, it can stop in the part to which a crystal is thin and the line defect generated in early stages of growth was extracted, and a good single crystal with few defects as a result is obtained.

[0011]

[Example] The manufacture approach of \*\*\*\* single crystal silicon carbide is concretely explained to an example based on drawing 1 and drawing 2.

[0012] First, one example of this invention is explained according to drawing 1. The SiC powder 4 as a raw material is put into the lower part of a crucible 3 like the approach mentioned above, and after covering with the lid of the crucible 3 with the electrode holder 1 which fixed the seed crystal 2 which becomes an underside from 6 H-SiC single crystal, this SiC powder 4 is made to heat and sublime by the RF creeping method of adjustment in this example.

[0013] And by making the above-mentioned seed crystal 2 into the low-temperature section, contact gasified SiC, it is made to recrystallize and the SiC single crystal 5 is grown up on seed crystal 2.

[0014] Now, the projection 7 for the crucible 3 used for this invention to once extract a crystal thinly near the front face of seed crystal 2, as shown in drawing 1 is formed in the crucible 3. With these projections 7, crystal growth is once extracted thinly, grows, is expanded again and grows. Consequently, it is stopped in the part into which a crystal is thin and the line defect 6 generated in early stages of growth was fastened, and a good SiC single crystal with few defects is obtained.

[0015] Thus, the defect density behind the formed SiC single crystal is about [ 103 //cm ] two, and the defect of the number of 10 is decreasing by about 1/by the approach shown in drawing 3 as compared with what carried out crystal growth, without once extracting thinly.

[0016] Drawing 2 is a sectional view of a crucible used for other examples of this invention. In this example, the heating apparatus 8 for making a part of crucible 3 into an elevated temperature near the front face of seed crystal 2 is formed.

[0017] Consequently, this seed crystal 2 neighborhood becomes an elevated temperature, the crystal growth in this neighborhood is barred, as a result, a crystal grows in the condition of having been extracted thinly, it expands again after that, and a crystal once grows.

[0018] Thus, by making it into an elevated temperature near [ a part of ] the front face of the seed crystal 2 of a crucible 3, the crystal growth of this neighborhood is barred, a crystal is once thinly extracted as a result, and it grows up. Consequently, it is stopped in the part into which a crystal is thin and the line defect 6 generated in early stages of growth was fastened, and a good single crystal with few defects is obtained.

[0019]

[Effect of the Invention] As explained above, according to this invention, it can stop in the part to which a crystal is thin and the line defect generated in early stages of growth was extracted, and a good single crystal with few defects is obtained. Therefore, the mass production nature of blue LED can be raised.

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TECHNICAL FIELD

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[Industrial Application] This invention relates to the manufacture approach of a silicon carbide single crystal, and relates to the approach of growing up a good single crystal with few defects especially.

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PRIOR ART

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EFFECT OF THE INVENTION

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[Effect of the Invention] As explained above, according to this invention, it can stop in the part to which a crystal is thin and the line defect generated in early stages of growth was extracted, and a good single crystal with few defects is obtained. Therefore, the mass production nature of blue LED can be raised.

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TECHNICAL PROBLEM

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[Problem(s) to be Solved by the Invention] When growing up a single crystal by the \*\*\*\* conventional approach, the 104 about [ $\mu\text{m} / 2$ ] consistency of a defect existed, and these defects had difficulties, such as having an adverse effect on the electrical characteristics of light emitting diode (LED).

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MEANS

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[Means for Solving the Problem]

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OPERATION

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[Function] According to this invention, it can stop in the part to which a crystal is thin and the line defect generated in early stages of growth was extracted, and a good single crystal with few defects as a result is obtained.

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EXAMPLE

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[Example] The manufacture approach of \*\*\*\* single crystal silicon carbide is concretely explained to an example based on drawing 1 and drawing 2.

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[0017] Consequently, this seed crystal 2 neighborhood becomes an elevated temperature, the crystal growth in this neighborhood is barred, as a result, a crystal grows in the condition of having been extracted thinly, it expands again after that, and a crystal once grows.

[0018] Thus, by making it into an elevated temperature near [ a part of ] the front face of the seed crystal 2 of a crucible 3, the crystal growth of this neighborhood is barred, a crystal is once thinly extracted as a result, and it grows up. Consequently, it is stopped in the part into which a crystal is thin and the line defect 6 generated in early stages of growth was fastened, and a good single crystal with few defects is obtained.

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DESCRIPTION OF DRAWINGS

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[Brief Description of the Drawings]

[Drawing 1] It is the sectional view showing one example of the crucible used for this invention.

[Drawing 2] It is the sectional view showing other examples of the crucible used for this invention.

[Drawing 3] It is the sectional view of a crucible used for the conventional single crystal growth.

[Description of Notations]

- 1 Electrode Holder
- 2 Seed Crystal
- 3 Crucible
- 4 Powder SiC
- 5 Single Crystal
- 7 Height
- 8 Heating Apparatus

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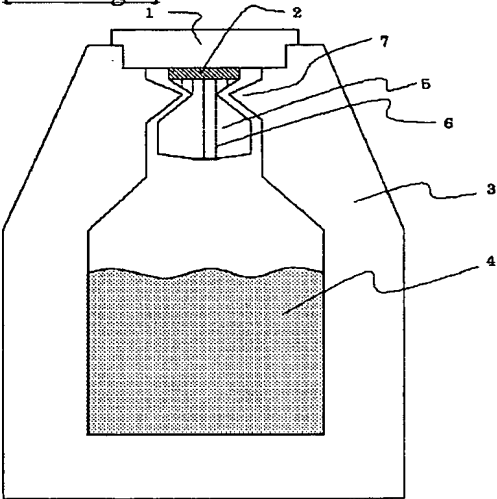
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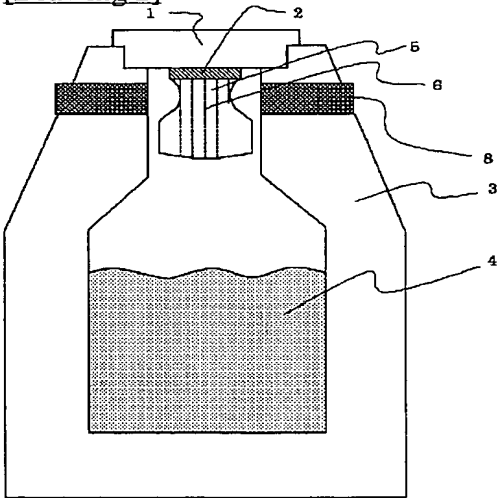
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DRAWINGS

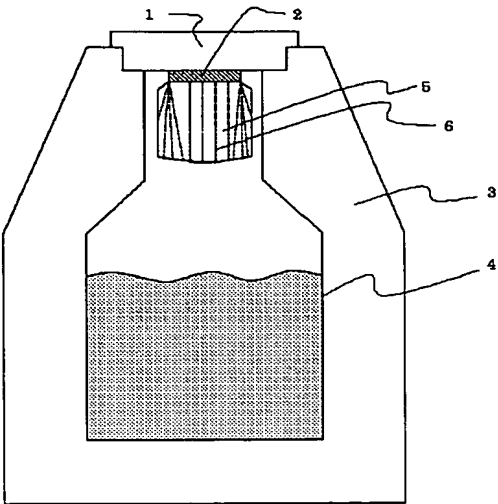
[Drawing 1]



[Drawing 2]



[Drawing 3]



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[Translation done.]

(51) Int.Cl.<sup>5</sup>

C 3 0 B 29/36

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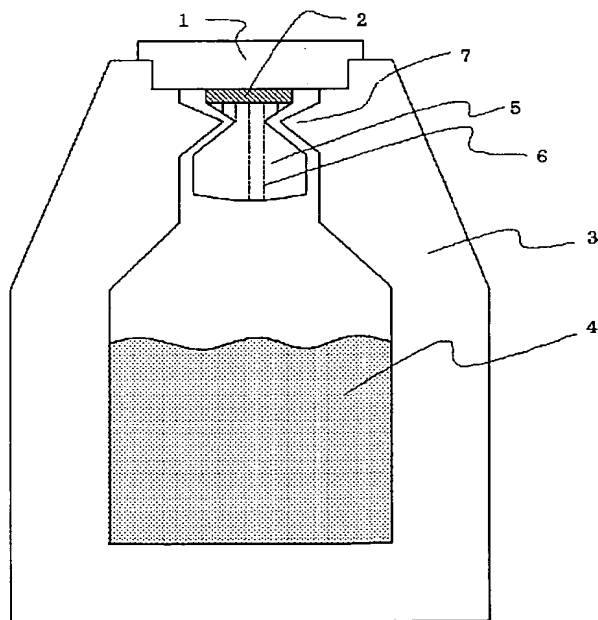
(74) 代理人 弁理士 鳥居 洋

(54) 【発明の名称】 単結晶炭化ケイ素の製造方法

(57) 【要約】

【目的】 この発明は、欠陥の少ない良質の単結晶を成長させる方法を提供することをその目的とする。

【構成】 この発明の単結晶炭化ケイ素の製造方法は、炭化ケイ素からなる原材料4を加熱昇華させ、炭化ケイ素単結晶からなる種結晶2上に供給し、この種結晶2上に炭化ケイ素単結晶6を成長させる方法において、結晶成長用ルツボ3の種結晶2近傍に突起物7を設け、この突起物で結晶を一旦細く絞って成長させ、再び拡大して炭化ケイ素単結晶6を成長させる。





## 【特許請求の範囲】

【請求項1】炭化ケイ素からなる原材料を加熱昇華させ、炭化ケイ素単結晶からなる種結晶上に供給し、この種結晶上に炭化ケイ素単結晶を成長させる方法において、結晶を一旦細く絞って成長させ、再び拡大することを特徴とする単結晶炭化ケイ素の製造方法。

【請求項2】結晶成長用ルツボ内の種結晶近傍に突起物を設け、結晶を一旦細く絞ることを特徴とする請求項1に記載の単結晶炭化ケイ素の製造方法。

【請求項3】結晶成長用ルツボ内の種結晶付近を高温にする手段を設け、結晶を一旦細く絞ることを特徴とする請求項1に記載の単結晶炭化ケイ素の製造方法。

## 【発明の詳細な説明】

## 【0001】

【産業上の利用分野】本発明は炭化ケイ素単結晶の製造方法に係り、特に、欠陥の少ない良質の単結晶を成長させる方法に関する。

## 【0002】

【従来の技術】炭化ケイ素(SiC)は耐熱性及び機械的強度も優れ、放射線に強いなどの物理的、化学的性質から耐環境性半導体材料として注目されている。特に6H形のSiC結晶は室温で約3eVの禁制帯幅を持ち、青色発光ダイオード材料として用いられている。

【0003】このようなSiC単結晶のインゴットを成長させる方法としては、例えば、「真空、30巻52～58頁(1987年)」に記載されているように、昇華法が採用されている。

【0004】図3に昇華法に用いられる従来の成長装置のルツボの断面図を示す。従来のSiCの結晶成長は6H-SiC単結晶からなる種結晶2を固着したホルダー1で蓋されるルツボ3の下部に、原材料としてのSiC粉末4を入れ、例えばルツボ3を高周波誘導法により加熱し、上記粉末SiC4を昇華させ、ガス状の原材料を上記種結晶2上に供給する。そして、上記種結晶2を低温部とすることでガス状の原材料が再結晶化し、上記種結晶2上に単結晶5が成長する。

【0005】このように結晶成長されたSiCには、図3に示すように、多数の線欠陥6が存在する。なお、図3に示す線欠陥6は模式的に示しており、実際は肉眼で見えるようなものではない。

## 【0006】

【発明が解決しようとする課題】斯る従来方法で単結晶を成長させた場合、欠陥の密度は $10^4$ 個/cm<sup>2</sup>程度存在し、これらの欠陥は発光ダイオード(LED)の電気的特性に悪影響を及ぼすなどの難点があった。

【0007】この発明は、上記事情に鑑みてなされたものにして、欠陥の少ない良質の単結晶を成長させる方法を提供することをその目的とする。

## 【0008】

【課題を解決するための手段】

【0009】この発明の単結晶炭化ケイ素の製造方法は、炭化ケイ素からなる原材料を加熱昇華させ、炭化ケイ素単結晶からなる種結晶上に供給し、この種結晶上に炭化ケイ素単結晶を成長させる方法において、結晶を一旦細く絞って成長させ、再び拡大することを特徴とする。

## 【0010】

【作用】本発明によれば、成長初期に発生した線欠陥を結晶の細く絞った部分で止めることができ、結果として欠陥の少ない良質の単結晶が得られる。

## 【0011】

【実施例】実施例に斯る単結晶炭化ケイ素の製造方法を図1及び図2に基づいて具体的に説明する。

【0012】まず、この発明の一実施例を図1に従い説明する。この実施例においては、前述した方法と同様にルツボ3の下部に原料としてのSiC粉末4を入れ、下面に6H-SiC単結晶からなる種結晶2を固着したホルダー1でルツボ3を蓋した後、例えば高周波誘導法によってこのSiC粉末4を加熱して昇華させる。

【0013】そして、上記種結晶2を低温部とすることにより、ガス化したSiCを接触させて再結晶させ、種結晶2上にSiC単結晶5を成長させるものである。

【0014】さて、この発明に用いられるルツボ3は、図1に示すように、種結晶2の表面近傍に、結晶を一旦細く絞るための突起物7がルツボ3内に設けられている。これらの突起物7により、結晶成長は一旦細く絞られて成長し、再び拡大し成長する。この結果、成長初期に発生した線欠陥6が結晶の細く絞められた部分で止められ、欠陥の少ない良質のSiC単結晶が得られる。

【0015】この様に形成したSiC単結晶後の欠陥密度は $10^3$ 個/cm<sup>2</sup>程度であり、図3に示す方法により、一旦細く絞らずに結晶成長したものに比して約1/10の数の欠陥が減少している。

【0016】図2はこの発明の他の実施例に用いられるルツボの断面図である。この実施例においては、種結晶2の表面近傍にルツボ3の一部を高温にするための加熱装置8が設けられている。

【0017】この結果、この種結晶2付近が高温になり、この付近における結晶成長が妨げられ、結果として一旦細く絞られた状態で結晶が成長し、その後、再び拡大して結晶が成長する。

【0018】この様に、ルツボ3の種結晶2の表面近傍の一部を高温にすることで、この付近の結晶成長が妨げられ、結果として、結晶が一旦細く絞られて成長する。この結果、成長初期に発生した線欠陥6が結晶の細く絞められた部分で止められ、欠陥の少ない良質な単結晶が得られる。

## 【0019】

【発明の効果】以上説明したように、この発明によれば、成長初期に発生した線欠陥を結晶の細く絞った部分

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で止めることができ、欠陥の少ない良質の単結晶が得られる。従って、青色LEDの量産性を高めることができる。

【図面の簡単な説明】

【図1】この発明に用いられるルツボの一実施例を示す断面図である。

【図2】この発明に用いられるルツボの他の実施例を示す断面図である。

【図3】従来の単結晶成長に用いられるルツボの断面図

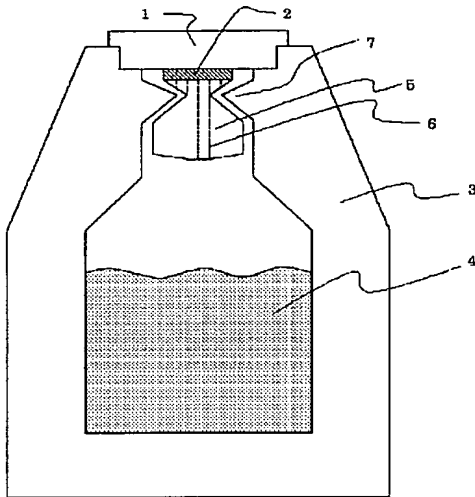
4

である。

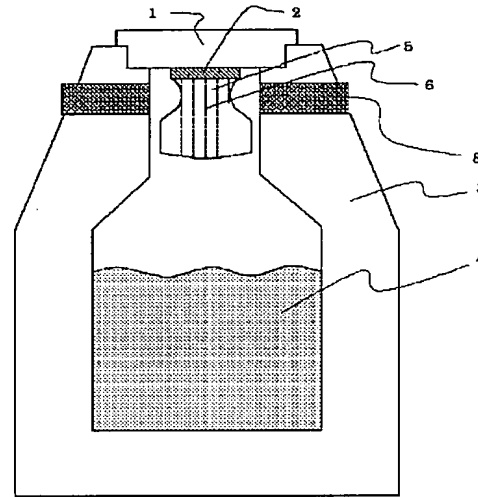
【符号の説明】

- 1 ホルダー
- 2 種結晶
- 3 ルツボ
- 4 粉末SiC
- 5 単結晶
- 7 突起部
- 8 加熱装置

【図1】



【図2】



【図3】

